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<p>(21) International Application Number: PCT/US00/12676</p> <p>(22) International Filing Date: 10 May 2000 (10.05.00)</p> <p>(30) Priority Data: 60/133,571 11 May 1999 (11.05.99) US 09/568,187 9 May 2000 (09.05.00) US</p> <p>(71) Applicant: PPG INDUSTRIES OHIO, INC. [US/US]; 3800 West 143rd Street, Cleveland, OH 44111 (US).</p> <p>(72) Inventors: MCCOLLUM, Gregory, J.; 5130 Bronwyn Court, Gibsonia, PA 15044 (US). HAUSER, Brian, T.; 4014 Chelstead Way, Murrysville, PA 15668 (US). VALKO, Joseph, T.; 420 Englewood Drive, Pittsburgh, PA 15237 (US). GRAY, Ralph, C.; 137 West Cruikshank Road, Butler, PA 16001 (US).</p> <p>(74) Agents: UHL, William, J.; PPG Industries, Inc., One PPG Place, Pittsburgh, PA 15272 (US) et al.</p>	<p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: PROCESS FOR THE TREATMENT OF CONTINUOUS COILS OF SHEET METAL AND PRODUCTS PREPARED THEREBY</p> <p>(57) Abstract</p> <p>An improved process for treating metal coated sheet steel such as galvanized sheet steel is provided. The process comprises the application of a Group IIIB or IVB metal compound to the surfaces of the metal coated sheet steel prior to contacting the metal surface with a protective lubricant.</p>		

\*-----BILLING INSTRUCTIONS-----\*

\*-----MATTER DESCRIPTION-----\*

U.S. TRADEMARK APPLN.  
SERIAL NO. 78/558597  
Mark: HIDEAWAY

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*-----CLIENT INFORMATION-----*
RGB SYSTEMS, INC.
JOINT BILL: 11845
BILL TEMPLATE: EJ FOLLOWUP TEMPLATE: ER
--CLIENT NUMBER-- --ORIGINATING-- --BILLING-- --SUPERVISING--
11845 JCH JCH JCH
J HOLMAN J HOLMAN J HOLMAN

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PHONE: 780-4088  
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PHONE:  
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STATUS:	OP	RATE:	1	ES
DATE OPENED:	02/03/2005	DEPT:	20	MIN FEE:
DATE CLOSED:		LOCATION:	01	MIN COST:
LAST RATE:	02/03/2005	PRACTICE:	1	MIN TOTAL:
HOLD FEES:		FEE FREQ:	M	FEE MARKUP: \$
HOLD COSTS:		COST FREQ:	M	COST MARKUP: \$
TRUST RET ACCT:		TRUST RETAIN:		FIXED COSTS: N
				FIXED FEES:
				CURRENCY: USD
				ARRANGEMENT: S
				MAXIMUM BILLINGS:

**TRADEMARK**

**Last Bill Date: 11/30/2005**

★-----TIME ENTRIES-----★

INDEX	INIT	DATE	STAT	HOURS	AMOUNT	BILLED		CUMULATIVE DESCRIPTION
80032	RSP	030306	B	.89	200.00			
	(RSP)							200.00
					FEE SUBTOTAL		200.00	

\*-----COST ENTRIES-----\*

INDEX	DATE	STAT	AMOUNT	DESCRIPTION	CODE	TKPR	VOUCHER
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262435	030306	B	1.00	Postage	POST	JCH	
				-----			
				2.00			

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EE	Estonia						

( ) BILL COSTS ONLY

( ) FINAL BILL

## PROCESS FOR THE TREATMENT OF CONTINUOUS COILS OF SHEET METAL AND PRODUCTS PREPARED THEREBY

## BACKGROUND OF THE INVENTION

10       The present invention relates to an improved process for the treatment of continuous coils of sheet metal produced in a steel mill.

          The production of light gauge steels for end uses ranging from architectural construction to automobiles is well known.

15       The coils of steel are produced on a cold rolling mill line to the preferred thickness and trimmed or slit to the required width. The metal may be oiled and used directly as cold rolled steel or it may be further coated with a relatively thin layer of a second metal on a continuous galvanizing line.

20       The galvanizing process may be performed in a bath of molten metal, typically with a zinc alloy, or it may be performed electrolytically with essentially pure zinc or with a co-deposit of zinc with a second metal such as iron. An annealing process may also be performed on the zinc coated

25       steel sheet to form an alloy with the steel substrate. The thus coated steel coil may then be oiled for storage or shipment to a customer for subsequent metal working operations. Eventually the oil must be removed by the customer using acid or alkaline cleaners before application of

30       passivating coatings ("pretreatment" coatings for adhesion and corrosion inhibition) or decorative coatings during manufacturing of the metal piece into a desired end product. Residues from the oil and cleaners may remain on the metal surface after cleaning and rinsing, inhibiting the active

35       ingredients in the passivating coatings from effectively bonding with metal atoms at the metal surface, thus diminishing the protective properties thereof.

251462	112305 B	3.00	Duplicating	DUPL	JCH
251463	112305 B	3.00	Postage	POST	JCH

6.00

BALANCE DUE FROM PREVIOUS STATEMENT 279.00  
LESS PAYMENT(S) (279.00)

BALANCE FORWARD 0.00

TIMECARD SUB-TOTAL ( .23) 60.00

DISBURSEMENT SUB-TOTAL 6.00

SUBTOTAL CURRENT PERIOD 66.00

TOTAL DUE 66.00

TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE 0.00

COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE 0.00

\*\*\*\*\* TIME AND FEE SUMMARY \*\*\*\*\*

TIMEKEEPER	RATE	HOURS	FEES
JOHN C. HOLMAN	400.00	.05	20.00
ROBERT S. PIERCE	222.22	.18	40.00
TOTALS		.23	60.00

\*\*\*\*\* COST CODE SUMMARY \*\*\*\*\*

COST CODE AMOUNT

DUPL Duplicating 3.00

POST Postage 3.00

COST TOTAL 6.00

\*\*\*\*\* LEDGER SUMMARY \*\*\*\*\*

Ledger Code	Ledger Description	Debit	Credit	Credit Applied To
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PAY Pay-Sntrst Oper 1451.50

COST Costs 412.50

FEES FEES 1019.00

FU-B FOLLOW-UP BILL 0.00

ZCOST Costs (by dept) 20.00

TOTAL 1451.50

412.50 COST

1019.00 FEES

20.00 ZCOST

AGED ACCOUNTS RECEIVABLE: 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES  
( ) BILL FEES ONLY

( ) DO NOT BILL  
( ) CLOSE FILE

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Hot dip galvanized sheet steel may also be passivated on the galvanizing line for resistance to white rust in storage, typically with a hexavalent chromium product. This is undesirable due to the toxicity of chromium, and the unsuitability as a base for subsequent coating.

It would be desirable to provide an improved process, without the use of chromium, for treating metals at the steel mill before application of protective lubricant, in order to enhance the corrosion protection of the metals as they are manufactured into end use products.

#### SUMMARY OF THE INVENTION

In accordance with the present invention, an improved process for treating a metal coated sheet steel is provided, the improvement comprising the following steps:

- a) contacting a surface of the metal coated sheet steel with a Group IIIB or IVB metal compound or mixture thereof dissolved or dispersed in a carrier medium; followed by
- b) contacting the surface of the metal coated sheet steel from step (a) with a protective lubricant.

Another aspect of the present invention is a treated metal coated sheet steel formed in accordance with the process outlined immediately above.

Another aspect of the present invention is a coated metal sheet steel comprising: (a) a metal substrate; (b) a coating comprising a Group IIIB or IVB metal compound or mixture thereof formed upon at least a portion of a surface of the metal substrate; and (c) a protective lubricant coating formed upon at least a portion of the coating.





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## DETAILED DESCRIPTION

Unless otherwise indicated, all numbers expressing quantities of ingredients or reaction conditions used in the specification and claims are to be understood as modified in  
5 all instances by the term "about".

The process of the present invention is typically used to produce sheet metals such as steel coated with zinc metal, zinc compounds or zinc alloys such as electrogalvanized steel, hot-dipped galvanized steel, Galvannealed steel, and steel  
10 plated with zinc alloy. Also, aluminum alloys, aluminum clad steel and aluminum alloy clad steel may be used. The shape of the metal substrate is an elongated strip wound about a spool in the form of a coil. The thickness of the strip preferably ranges from about 0.254 to about 3.18 millimeters (mm) (about  
15 10 to about 125 mils), and more preferably about 0.3 mm, although the thickness can be greater or less, as desired. The width of the strip generally ranges from about 30.5 to about 183 centimeters (about 12 to about 72 inches), although the width can vary depending upon its intended use.

20 After coating the steel substrate, such as by electro galvanizing or hot dip galvanizing, and cooling the metal surface typically to a temperature of 100-300°F (37-149°C) and prior to the application of a protective lubricant such as oiling of the metal, the surface of the sheet metal is  
25 contacted with a Group IIIB or IVB metal compound.

The Group IIIB or IVB metal compound is typically dispersed or dissolved in a carrier medium such as a aqueous medium, vapor, or aerosol. The solution or dispersion may be applied to the metal at a carrier medium temperature of about  
30 ambient to 150°F (ambient to 65°C) by known application techniques, such as roll coating, dipping or immersion, spraying, intermittent spraying, dipping followed by spraying or spraying followed by dipping. The vapor or aerosol containing the Group IIIB or IVB metal may be applied by  
35 passing the continuous strip of metal through a chamber in which the vapor is present. Treatment times are typically

BALANCE DUE FROM PREVIOUS STATEMENT	152.75
LESS PAYMENT(S)	(152.75)
BALANCE FORWARD	0.00
TIMECARD SUB-TOTAL ( 1.60) 280.00	
DISBURSEMENT SUB-TOTAL	0.50
SUBTOTAL CURRENT PERIOD	280.50
TOTAL DUE	280.50
TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE 0.00	
COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE	0.00

\*-----TIME AND FEE SUMMARY-----\*

*-----TIMEKEEPER-----*	RATE	HOURS	FEE
ROBERT S. PIERCE	175.00	1.60	100.00
TOTALS	1.60		280.00

\*-----COST CODE SUMMARY-----\*

*-----COST CODE-----*	AMOUNT
DUPL Duplicating	0.25
POST Postage	0.25
COST TOTAL	0.50

\*-----LEDGER SUMMARY-----\*

Ledger Code	Ledger Description	Debit	Credit	Credit Applied To
PAY	Pay-Sntrst Oper	996.50		
				166.50 COST
				810.00 FEES
				20.00 ZCOST
COST	Costs	166.50		
FEES	FEES	810.00		
ZCOST	Costs (by dept)	20.00		
TOTAL		996.50	996.50	

AGED ACCOUNTS RECEIVABLE: 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES ( ) DO NOT BILL  
( ) BILL FEES ONLY ( ) CLOSE FILE  
( ) BILL COSTS ONLY ( ) FINAL BILL

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short, from a few seconds up to a minute, depending on the speed of the sheet metal line, and the type of coater being used.

The Group IIIB or IVB metals referred to herein are those elements included in such groups in the CAS Periodic Table of the Elements as is shown, for example, in the *Handbook of Chemistry and Physics*, 63rd Edition (1983).

Preferred Group IIIB and IVB metal compounds are compounds of zirconium, titanium, hafnium, yttrium and cerium and mixtures thereof. Typical zirconium compounds may be selected from hexafluorozirconic acid, alkali metal and ammonium salts thereof, ammonium zirconium carbonate, zirconyl nitrate, zirconium carboxylates and zirconium hydroxy carboxylates such as hydrofluorozirconic acid, zirconium acetate, zirconium oxalate, ammonium zirconium glycolate, ammonium zirconium lactate, ammonium zirconium citrate, and mixtures thereof. Hexafluorozirconic acid is preferred. An example of an yttrium compound is yttrium nitrate. An example of the titanium compound is fluorotitanic acid and its salts. An example of the hafnium compound is hafnium nitrate. An example of the cerium compound is cerous nitrate. The Group IIIB or IVB metal compound is present in the carrier medium in an amount of 10 to 5000 ppm metal, preferably 100 to 1000 ppm metal.

For aqueous carriers, the pH of the solution with dispersion usually ranges from 2.0 to about 7.0 and is preferably about 3.5 to about 5.5. The pH may be adjusted using mineral acids such as hydrofluoric acid, fluoroboric acid, phosphoric acid, and the like, including mixtures thereof; organic acids such as lactic acid, acetic acid, citric acid, or mixtures thereof; and water soluble or water dispersible bases such as sodium hydroxide, ammonium hydroxide, ammonia, or amines such as triethylamine, methylethyl amine, or mixtures thereof.

In one embodiment of the invention, the solution or dispersion further contains a film forming resin. Suitable

\*-----BILLING INSTRUCTIONS-----\*

\*-----MATTER DESCRIPTION-----\*  
U.S. REGISTRATION NO. 2937056  
MARK: IP LINK

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11845  
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FOLLOWUP TEMPLATE: ER JCH  
JCH  
J HOLMAN

BILL TEMPLATE: EJ

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STATUS: OP RATE: 1  
DATE OPENED: 07/29/2004 DEPT: 20  
DATE CLOSED: LOCATION: 01  
LAST RATE: 07/29/2004 PRACTICE: 1  
HOLD FEES: FEE FREQ: M  
HOLD COSTS: COST FREQ: M  
TRUST RET ACCT: TRUST RETAIN:

MIN FEE:  
MIN COST:  
MIN TOTAL:  
FEE MARKUP: \$  
COST MARKUP: \$  
FIXED FEES:  
CURRENCY: USD  
MAXIMUM BILLINGS:

TRADEMARK

Last Bill Date: 05/26/2005

\*-----TIME ENTRIES-----\*

INDEX	INIT	DATE	STAT	HOURS	AMOUNT	BILLED	CUMULATIVE DESCRIPTION
77148	RSP	021306	B	.60	105.00	.60	105.00 REVIEW PATTON RESPONSE TO DEMAND LETTER; (RSP) LETTER TO CLIENT REPORTING SAME
77155	RSP	022206	B	1.00	175.00	1.00	280.00 REVIEW PATTON LINKS; LETTER TO PATTON; LETTER (RSP) TO CLIENT
FEE SUBTOTAL					280.00		

\*-----COST ENTRIES-----\*

INDEX	DATE	STAT	AMOUNT	DESCRIPTION	CODE	TKPER	VOUCHER
265878	032106	B	0.25	Duplicating	DUPL	JCH	
265879	032106	B	0.25	Postage	POST	JCH	

0.50

- 5 -

resins include reaction products of one or more alkanolamines and an epoxy-functional material containing at least two epoxy groups, such as those disclosed in U. S. Patent 5,653,823. Preferably, such resins contain beta hydroxy ester, imide, or sulfide functionality, incorporated by using

5 dimethylolpropionic acid, phthalimide, or mercaptoglycerine as an additional reactant in the preparation of the resin. Other suitable resins include water soluble and water dispersible polyacrylic acids as disclosed in U. S. Patents 3,912,548 and

10 5,328,525; phenol-aldehyde resins as described in U. S. Patent 5,662,746; water soluble polyamides such as those disclosed in WO 95/33869; copolymers of maleic or acrylic acid with allyl ether as described in Canadian Patent application 2,087,352; and water soluble and dispersible resins including epoxy

15 resins, aminoplasts, phenol-formaldehyde resins, tannins, and polyvinyl phenols as discussed in U. S. Patent 5,449,415. Another suitable resin is the reaction product of an epoxy-containing polymer or oligomer (polyepoxide) with a dialkanolamine and a hydroxy acid. Examples of suitable

20 epoxy-containing polymers or oligomers include polyglycidyl ethers of polyhydric phenols such as the polyglycidyl ether of Bisphenol A. The preferred polyglycidyl ether is the diglycidyl ether of Bisphenol A.

Examples of dialkanolamines include those which contain

25 up to four carbon atoms such as diisopropanolamine, diethanolamine, di(2-hydroxybutyl)amine, and N-(3-hydroxypropyl)ethanolamine. Diethanolamine is preferred.

Examples of hydroxy acids include dimethylolpropionic acid, which is preferred, trimethylolpropionic acid,

30 pentaerythritol, malic acid, lactic acid, glycolic acid, gluconic acid, glucuronic acid, citric acid, 3-hydroxypivalic acid, and salicylic acid.

The reaction product may be prepared as follows: the epoxy-containing polymer or oligomer is added to a suitable

35 reaction vessel with an organic solvent. Suitable solvents include glycol ethers such as ethylene glycol methyl ether and

TOTAL DUE . . . . . 1.54

TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE . . . . . 0.00

COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE . . . . . 0.00

\*-----COST CODE SUMMARY-----\*

*-----COST CODE-----*	AMOUNT
DUPL Duplicating	0.50
POST Postage	0.50
TELE Telephone	0.54
<b>COST TOTAL</b>	<b>1.54</b>

\*-----LEDGER SUMMARY-----\*

Ledger Code	Ledger Description	Debit	Credit	Credit Applied To
PAY	Pay-Sntst Oper		1945.00	
				525.50 COST
				1384.00 FEES
				35.50 ZCOST
COST	Costs	525.50		
FEES	FEES	1384.00		
FU-B	FOLLOW-UP BILL	0.00		
ZCOST	Costs (by dept)	35.50		
<b>TOTAL</b>		<b>1945.00</b>	<b>1945.00</b>	

AGED ACCOUNTS RECEIVABLE: 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES ( ) DO NOT BILL

( ) BILL FEES ONLY ( ) CLOSE FILE

( ) BILL COSTS ONLY ( ) FINAL BILL

- 6 -

propylene glycol methyl ether. The mixture is heated to a temperature of about 50°C, the amine and hydroxy acid are added, and the mixture allowed to exotherm to a temperature of about 90 to 100°C.

5       The mole ratio of the epoxy-containing polymer or oligomer to hydroxy acid to amine is from 0.6 to 5.0:0.05 to 5.5:1, preferably 1.5 to 2.5:1.0 to 2.0:1.

      The reaction product may then be thinned with additional organic solvents and/or water preferably containing an acid  
10   which can form the carrier medium for the reaction product. Examples of other organic solvents include alcohols with up to about 8 carbon atoms such as methanol, isopropanol, and the like, additional glycol ethers such as the monoalkyl ethers of ethylene glycol, diethylene glycol, or propylene glycol, and  
15   the like. Water, containing sulfamic acid, is the preferred ingredient in the carrier medium. When present, the water dispersible organic solvents are typically used in amounts up to about twenty (20) percent, preferably ten (10) percent by volume, based on the total volume of the carrier medium with  
20   water being the remaining.

      When present, the film forming resinous binder is in the carrier medium of the present invention in an amount of 0.005% to 30% based on the total weight of the ingredients in the medium, and the weight ratio of the resin to Group IIIB or IVB  
25   metal or metal compound is from 2.0 to 10.0:1, preferably 3.0 to 5.0:1 based on metal.

      Other optional materials in the carrier medium include surfactants that function as defoamers or substrate wetting agents. Anionic, cationic, amphoteric, or nonionic  
30   surfactants may be used. Compatible mixtures of such materials are also suitable. Surfactants are typically present at levels up to about 1 percent, preferably up to about 0.1 percent by volume, and wetting agents are typically present at levels up to about 2 percent, preferably up to  
35   about 0.5 percent by volume, based on the total volume of carrier medium.

\*-----BILLING INSTRUCTIONS-----\*

\*-----MATTER DESCRIPTION-----\*  
U.S. Trademark Application  
Serial No. 78/534200  
Mark: EXTRON

\*-----CLIENT INFORMATION-----\*  
RGB SYSTEMS, INC. --CLIENT NUMBER-- --ORIGINATING-- --BILLING-- --SUPERVISING--  
11845 JCH JCH JCH

JOINT BILL: 11845 BILL TEMPLATE: EJ FOLLOWUP TEMPLATE: ER

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REFERRED BY:

STATUS: OP RATE: 1  
DATE OPENED: 12/17/2004 DEPT: 20  
DATE CLOSED: LOCATION: 01  
LAST RATE: 07/21/2004 PRACTICE: 1  
HOLD FEES: FEE FREQ: M  
HOLD COSTS: COST FREQ: M  
TRUST RET ACCT: TRUST RETAIN:  
MIN FEE: ES  
MIN COST: 0  
MIN TOTAL: 0  
FEE MARKUP: \$  
COST MARKUP: \$  
FIXED FEES: N  
CURRENCY: USD  
MAXIMUM BILLINGS:

TRADEMARK

Last Bill Date: 03/22/2006

\*-----COST ENTRIES-----\*

INDEX	DATE STAT	AMOUNT	DESCRIPTION	CODE	TKPER	VOUCHER
262251	020706 B	0.54	Telephone	tele	RSP	
266980	032706 B	0.50	Duplicating	DUPL	JCH	
266981	032706 B	0.50	Postage	POST	JCH	

1.54

BALANCE DUE FROM PREVIOUS STATEMENT 114.50  
LESS PAYMENT(S) (114.50)

BALANCE FORWARD 0.00

TIMECARD SUB-TOTAL ( .00) 0.00  
DISBURSEMENT SUB-TOTAL 1.54  
SUBTOTAL CURRENT PERIOD 1.54



- 7 -

The film coverage of the residue of the pretreatment coating composition generally ranges from about 1 to about 1000 milligrams per square meter ( $\text{mg}/\text{m}^2$ ), and is preferably about 10 to about 400  $\text{mg}/\text{m}^2$ . The thickness of the pretreatment  
5 coating can vary, but is generally less than about 1 micrometer, preferably ranges from about 1 to about 500 nanometers, and more preferably is about 10 to about 300 nanometers.

After contact with the Group IIIB or IVB metal compound,  
10 the sheet metal is coated with a protective wet or dry lubricant for shipping or storage. Such lubricants may be any of those used conventionally in the art. An example of a wet lubricant is oil. U. S. Patent No. 5,229,450 describes a suitable dry lubricant.

15 Not intending to be bound by any theory, the inventors believe that by forming the metal according to the process of the present invention, a stronger chemical interaction between metal atoms at the metal surface and the Group IIIB or IVB metal compound is possible because the virgin metal surface is  
20 free of residues of oil and alkaline or acidic cleaners that may inhibit chemical reaction, thus providing enhanced corrosion protection of the finished metal product.

BALANCE DUE FROM PREVIOUS STATEMENT	226.00
LESS PAYMENT(S)	(226.00)
BALANCE FORWARD	0.00
TIMECARD SUB-TOTAL ( 1.15)	310.00
DISBURSEMENT SUB-TOTAL	0.34
SUBTOTAL CURRENT PERIOD	310.34
TOTAL DUE	310.34
TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE	0.00
COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE	265.50

\*\*\*\*\*TIME AND FEE SUMMARY\*\*\*\*\*

TIMEKEEPER	RATE	HOURS	%	FEES	%
JOHN C. HOLMAN	423.08	.26	22.6	110.00	35.5
ROBERT S. PIERCE	224.72	.89	77.4	200.00	64.5
TOTALS		1.15		310.00	

\*\*\*\*\*COST CODE SUMMARY\*\*\*\*\*

COST CODE	AMOUNT
TELE Telephone	0.34
COST TOTAL	0.34

\*\*\*\*\*LEDGER SUMMARY\*\*\*\*\*

Ledger Code	Ledger Description	Debit	Credit	Credit Applied To
PAY	Pay-Sntstet Oper		1608.00	
				869.00 COST
				729.00 FEES
				10.00 ZCOST
COST	Costs	869.00		
FEES	FEES	729.00		
ZCOST	Costs (by dept)	10.00		
TOTAL		1608.00	1608.00	

AGED ACCOUNTS RECEIVABLE: 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES ( ) DO NOT BILL  
 ( ) BILL FEES ONLY ( ) CLOSE FILE  
 ( ) BILL COSTS ONLY ( ) FINAL BILL

- 8 -

## WE CLAIM:

1. In a process for treating a metal coated steel sheet, the improvement comprising the following steps:
  - 5 (a) contacting a surface of the metal coated sheet steel with a Group IIIB or IVB metal compound or mixture thereof dissolved or dispersed in a carrier medium; followed by
  - (b) contacting the surface of the metal coated sheet steel from step (a) with a protective lubricant.
- 10 2. The process of claim 1 wherein the Group IIIB or IVB metal compound is present in an aqueous carrier medium, vapor or aerosol.
- 15 3. The process of claim 2 wherein the metal surface is contacted with the Group IIIB or IVB metal compound in the aqueous carrier medium at a carrier medium temperature of about ambient to 150°F (ambient to 65°C).
- 20 4. The process of claim 2 wherein the metal surface is contacted with the Group IIIB or IVB metal compound by roll coating.
- 25 5. The process of claim 2 wherein the metal surface is contacted with the Group IIIB or IVB metal compound as an aerosol or a vapor.
- 30 6. The process of claim 1 wherein the Group IIIB or IVB metal compound is a zirconium compound.
7. The process of claim 6 wherein the zirconium compound is present in the aqueous medium in an amount of 10 to 5000 ppm Zr.
- 35 8. The process of claim 2 wherein the aqueous medium further comprises a resinous binder.

\*-----BILLING INSTRUCTIONS-----\*

\*-----MATTER DESCRIPTION-----\*

Taiwanese Trademark Appln. No. 94026676  
Mark: EXTRON

\*-----CLIENT INFORMATION-----\*

RGB SYSTEMS, INC.

--CLIENT NUMBER--

11845

--BILLING--

JCH

--SUPERVISING--

JCH

JOINT BILL: 11845

BILL TEMPLATE: EJ FOLLOWUP TEMPLATE: ER

J HOLMAN

\*-----CLIENT ADDRESS-----\*

RGB SYSTEMS, INC.  
1230 SOUTH LEWIS STREET  
ANAHEIM, CA 92805

\*-----MATTER ADDRESS-----\*

\*-----MATTER INFORMATION-----\*

PHONE: 780-4088

REFERRED BY:

PHONE:

CONTACT:

STATUS: OP

RATE:

1

TEMPLATE: ES

MIN FEE:

DATE OPENED: 06/01/2005

DEPT: 20

TIME FORMAT: 0

MIN COST:

DATE CLOSED:

LOCATION: -01

COST FORMAT: 0

MIN TOTAL:

LAST RATE: 07/21/2004

PRACTICE: 1

INTRST CODE: 0

FEE MARKUP: %

HOLD FEES:

FEE FREQ: M

INT FREE DAY:

COST MARKUP: %

HOLD COSTS:

COST FREQ: M

FIXED COSTS: N

FIXED FEES:

TRUST RET ACCT:

TRUST RETAIN:

ARRANGEMENT: S

CURRENCY: USD

MAXIMUM BILLINGS:

TRADEMARK

01/01/00

.00

.00

Last Bill Date: 10/20/2005

\*-----TIME ENTRIES-----\*

				WORKED	BILLED		
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80024	JCH	030606	B	.26	110.00	110.00	FOR REPORTING REGISTRAR OFFICE ACTION; EMAILS WITH TAIWAN ASSOCIATE REGARDING RESPONSE AND EXTENSION; REPORTING EXTENSION OF TIME
				(JCH)			
80033	RSP	030606	B	.89	200.00	310.00	
				(RSP)			
				FEE SUBTOTAL	310.00		

\*-----COST ENTRIES-----\*

INDEX	DATE	STAT	AMOUNT	DESCRIPTION	CODE	TKPER	VOUCHER
237317	010306	B	0.34	Telephone	tele		RSP
				0.34			

- 9 -

9. The process of claim 8 wherein the resinous binder comprises a reaction product of an epoxy group-containing polymer or oligomer, a hydroxy functional acid and a dialkanolamine.

10. The process of claim 9 wherein the epoxy group-containing polymer or oligomer is a polyglycidyl ether of a polyhydric phenol.

11. The process of claim 9 wherein the epoxy group-containing polymer or oligomer is the diglycidyl ether of Bisphenol A.

12. The process of claim 9 in which the dialkanolamine is diethanolamine.

13. The process of claim 9 in which the hydroxy functional acid is dimethylolpropionic acid.

14. The process of claim 9 wherein the epoxy group-containing polymer or oligomer, hydroxy functional acid, and dialkanolamine are reacted in a 0.6 to 5.0:0.05 to 5.5:1 mole ratio.

15. The process of claim 9 wherein the medium is substantially free of chrome.

16. The process of claim 1 wherein the metal is zinc coated steel.

17. A treated metal coated sheet steel formed in accordance with the process of claim 1.

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BALANCE DUE FROM PREVIOUS STATEMENT	294.00
LESS PAYMENT(S)	(294.00)
BALANCE FORWARD	0.00
TIMECARD SUB-TOTAL ( .51)	140.00
DISBURSEMENT SUB-TOTAL	0.50
SUBTOTAL CURRENT PERIOD	140.50
TOTAL DUE	140.50
TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE	0.00
COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE	0.00

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*-----TIME AND FEE SUMMARY-----*			
TIMEKEEPER	RATE	HOURS	FEE
JOHN C. HOLMAN	423.08	.13	55.00
ROBERT S. PIERCE	223.68	.38	85.00
TOTALS		.51	140.00

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*-----COST CODE SUMMARY-----*	
COST CODE	AMOUNT
DUPL Duplicating	0.25
POST Postage	0.25
COST TOTAL	0.50

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*-----LEDGER SUMMARY-----*			
Ledger Code	Ledger Description	Debit	Credit
PAY	Pay-Sntrst Oper	1855.00	
			1003.00 COST
			837.00 FEES
			15.00 ZCOST
COST	Costs	1003.00	
FEES	FEES	837.00	
ZCOST	Costs (by dept)	15.00	
TOTAL		1855.00	1855.00

AGED ACCOUNTS RECEIVABLE: 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES  
( ) BILL FEES ONLY  
( ) BILL COSTS ONLY  
( ) DO NOT BILL  
( ) CLOSE FILE  
( ) FINAL BILL

- 10 -

18. A coated metal sheet steel, comprising:
- (a) a metal substrate;
  - (b) a coating comprising a Group IIIB or IVB metal compound or mixture thereof formed upon at least a portion of  
5 a surface of the metal substrate;
  - (c) and a protective lubricant coating formed upon at least a portion of the coating.
19. The coated metal sheet steel of claim 18 wherein the  
10 Group IIIB or IVB metal compound is a zirconium compound.
20. The coated metal sheet steel of claim 18 wherein the  
coating comprising a Group IIIB or IVB metal compound or  
mixture thereof is substantially free of chrome.  
15
21. The coated metal sheet steel of claim 18 wherein the  
metal is zinc coated steel.

\*-----BILLING INSTRUCTIONS-----\*

\*-----MATTER DESCRIPTION-----\*

New Zealand Trademark Application

Mark: EXTRON

```

*-----CLIENT INFORMATION-----*
RGB SYSTEMS, INC.
11845
--CLIENT NUMBER--
11845
--ORIGINATING--
JCH
--BILLING--
JCH
--SUPERVISING--
J HOLMAN
J HOLMAN
BILL TEMPLATE: EJ FOLLOWUP TEMPLATE: ER
JOINT BILL: 11845

```

★-----MATTER ADDRESS-----★

\*-----CLIENT ADDRESS-----\*

RGB SYSTEMS, INC.  
1230 SOUTH LEWIS STREET  
ANAHEIM, CA 92805

\*-----MATTER INFORMATION-----\*

PHONE:  
CONTACT:

STATUS:	OP	RATE:	1	TEMPLATE:	ES	MIN FEE:
DATE OPENED:	06/01/2005	DEPT:	20	TIME FORMAT:	0	MIN COST:
DATE CLOSED:		LOCATION:	01	COST FORMAT:	0	MIN TOTAL:
LAST RATE:	07/21/2004	PRACTICE:	1	INTREST CODE:	0	FEE MARKUP: %
HOLD FEES:		FEE FREQ:	M	INT FREE DAY:		COST MARKUP: %
HOLD COSTS:		COST FREQ:	M	FIXED COSTS:	N	FIXED FEES:
TRUST RET ACCT:		TRUST RETAIN:		ARRANGEMENT:	S	CURRENCY: USD
						MAXIMUM BILLINGS:

TRADEMARK	01/01/00	.00
	01/01/00	.00

**Last Bill Date: 11/30/2005**

★-----TIME ENTRIES-----★

WORKED				BILLED		
INDEX	INIT	DATE	STAT	HOURS	AMOUNT	CUMULATIVE DESCRIPTION
80027	JCH	031406	B	.13	55.00	55.00 FOR FORWARDING NEW ZEALAND REGISTRATION
	(JCH)					CERTIFICATES POLEARAY AND POLESURROUND;
						REQUISITE DOCKETING
80030	RSP	031406	B	.38	85.00	140.00
	(RSP)					
FEE SUBTOTAL						140.00

★-----COST ENTRIES-----★

INDEX	DATE	STAT	AMOUNT	DESCRIPTION	CODE	TKPER	VOUCHER
263815	031006	B	0.25	Duplicating	DUPL	JCH	
263816	031006	B	0.25	Postage	POST	JCH	

0.50



# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 00/12676

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 C23C22/36 C23C28/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 95 02660 A (HENKEL CORP) 26 January 1995 (1995-01-26) page 16, line 32 -page 17, line 9; claim 1	1-21
X	US 5 584 944 A (BERSHAS JAMES P ET AL) 17 December 1996 (1996-12-17) column 11, line 34 - line 47 column 1, line 26 - line 40	1-21
A	US 5 801 217 A (MURPHY JOSEPH E ET AL) 1 September 1998 (1998-09-01)  column 5, line 22 - line 67; claim 1	1,2, 8-11, 19-21

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"A" document member of the same patent family

Date of the actual completion of the international search

12 September 2000

Date of mailing of the international search report

16/10/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
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Authorized officer

De Anna, P

TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE . . . . . 0.00  
COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE . . . . . 0.00

*-----COST CODE SUMMARY-----*		AMOUNT
*-----COST CODE-----*		
FA Foreign Associates		272.73
COST TOTAL		272.73

*-----LEDGER SUMMARY-----*			
Ledger Code	Ledger Description	Debit	Credit
PAY	Pay-Sntrst Oper	1837.00	
			1062.00 COST
			760.00 FEES
			15.00 ZCOST
COST	Costs	1062.00	
FEES	FEES	760.00	
ZCOST	Costs (by dept)	15.00	
TOTAL		1837.00	1837.00

AGED ACCOUNTS RECEIVABLE: . . . 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES  
( ) BILL FEES ONLY  
( ) BILL COSTS ONLY  
( ) DO NOT BILL  
( ) CLOSE FILE  
( ) FINAL BILL

# INTERNATIONAL SEARCH REPORT

Information on patent family members

In International Application No  
PCT/US 00/12676

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9502660 A	26-01-1995	US 5378379 A	03-01-1995
		US 5458698 A	17-10-1995
		US 5476601 A	19-12-1995
		AU 707266 B	08-07-1999
		AU 5036498 A	26-03-1998
		AU 683047 B	30-10-1997
		AU 6082094 A	13-02-1995
		BR 9407122 A	10-09-1996
		CN 1127010 A, B	17-07-1996
		CN 1203265 A	30-12-1998
		EP 0708812 A	01-05-1996
		JP 9500408 T	14-01-1997
		PL 312564 A	29-04-1996
		US 5486316 A	23-01-1996
US 5584944 A	17-12-1996	US 5584944 A	17-12-1996
		US 5476601 A	19-12-1995
		US 5458698 A	17-10-1995
		US 5080814 A	14-01-1992
		US 4944889 A	31-07-1990
		US 4859351 A	22-08-1989
		AU 707266 B	08-07-1999
		AU 5036498 A	26-03-1998
		AU 683047 B	30-10-1997
		AU 6082094 A	13-02-1995
		BR 9407122 A	10-09-1996
		CN 1127010 A, B	17-07-1996
		CN 1203265 A	30-12-1998
		EP 0708812 A	01-05-1996
		JP 9500408 T	14-01-1997
		PL 312564 A	29-04-1996
		WO 9502660 A	26-01-1995
		US 5486316 A	23-01-1996
		AU 675800 B	20-02-1997
		AU 4665493 A	31-01-1994
		BR 9306696 A	08-12-1998
		CA 2135920 A	20-01-1994
		CN 1085244 A	13-04-1994
		EP 0649458 A	26-04-1995
		EP 0969078 A	05-01-2000
		JP 7509261 T	12-10-1995
		MD 960241 A	30-04-1998
		MX 9304090 A	31-03-1994
		WO 9401517 A	20-01-1994
		US 5389199 A	14-02-1995
		US 5584943 A	17-12-1996
		ZA 9304846 A	03-02-1994
		AT 111777 T	15-10-1994
		AU 629675 B	08-10-1992
		AU 6110790 A	21-02-1991
		BR 9004082 A	03-09-1991
		CN 1052136 A, B	12-06-1991
		DE 69012713 D	27-10-1994
		DE 69012713 T	23-02-1995
		EP 0413328 A	20-02-1991
		GR 3024135 T	31-10-1997
		HK 1007700 A	23-04-1999
		KR 159771 B	18-02-1999

\*-----BILLING INSTRUCTIONS-----\*

\*-----MATTER DESCRIPTION-----\*  
NEW CANADIAN TRADEMARK APPLICATION  
MARK: EXTRON

\*-----CLIENT INFORMATION-----\*  
RGB SYSTEMS, INC.  
JOINT BILL: 11845  
BILL TEMPLATE: EJ FOLLOWUP TEMPLATE: ER  
--CLIENT NUMBER-- 11845  
--ORIGINATING-- JCH JCH  
--BILLING-- JCH JCH  
--SUPERVISING-- JCH JCH

\*-----CLIENT ADDRESS-----\*  
RGB SYSTEMS, INC.  
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ANAHEIM, CA 92805  
\*-----MATTER INFORMATION-----\*  
PHONE: 780-4088  
REFERRED BY:  
\*-----MATTER ADDRESS-----\*  
CONTACT:  
PHONE:

STATUS: OP RATE: 1  
DATE OPENED: 06/01/2005 DEPT: 20  
DATE CLOSED: LOCATION: 01  
LAST RATE: 07/21/2004 PRACTICE: 1  
HOLD FEES: FEE FREQ: M  
HOLD COSTS: COST FREQ: M  
TRUST RET ACCT: TRUST RETAIN: S  
MIN FEE: ES  
MIN COST: 0  
MIN TOTAL: 0  
FEE MARKUP: %  
COST MARKUP: %  
FIXED FEES: N  
ARRANGEMENT: S  
CURRENCY: USD  
MAXIMUM BILLINGS:

TRADEMARK  
01/01/00 01/01/00 .00 .00

Last Bill Date: 01/23/2006

\*-----COST ENTRIES-----\*

INDEX	DATE STAT	AMOUNT	DESCRIPTION	CODE	TXPER VOUCHER
233240	011706 B	272.73	Foreign Associates - - VENDOR:RIDOUT & MAYBEE LLP	FA	JCH 108842

BALANCE DUE FROM PREVIOUS STATEMENT . . . . . 365.82  
LESS PAYMENT(S) . . . . . (365.82)  
BALANCE FORWARD . . . . . 0.00  
TIMECARD SUB-TOTAL ( .00) . . . . . 0.00  
DISBURSEMENT SUB-TOTAL . . . . . 272.73  
SUBTOTAL CURRENT PERIOD . . . . . 272.73  
TOTAL DUE . . . . . 272.73

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Int'l. Application No.

PCT/US 00/12676

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5584944 A		US 5030323 A	09-07-1991
		US 5064500 A	12-11-1991
		AT 91145 T	15-07-1993
		AT 136578 T	15-04-1996
		AU 606690 B	14-02-1991
		AU 1694888 A	01-12-1988
		BR 8802629 A	27-12-1988
		CN 1030245 A, B	11-01-1989
US 5801217 A	01-09-1998	US 5693371 A	02-12-1997
		CA 2251374 A	23-04-1998
		EP 0932453 A	04-08-1999
		WO 9816324 A	23-04-1998

TIME VALUE FOR THE MATTER AFTER THE CUTOFF DATE . . . . . 0.00  
COST VALUE FOR THE MATTER AFTER THE CUTOFF DATE . . . . . 0.00

*-----TIME AND FEE SUMMARY-----*				
TIMEKEEPER	RATE	HOURS	%	FEE
JOHN C. HOLMAN	416.67	.42	22.6	175.00 35.0
ROBERT S. PIERCE	225.69	1.44	77.4	325.00 65.0
TOTALS		1.86		500.00

*-----LEDGER SUMMARY-----*				
Ledger Code	Ledger Description	Debit	Credit	Credit Applied To
PAY	Pay-Sntrst Oper		11230.72	
COST	Costs	2023.22		2023.22 COST
FEE	FEE	9112.50		9112.50 FEES
ZCOST	Costs (by dept)	95.00		95.00 ZCOST
TOTAL		11230.72	11230.72	

AGED ACCOUNTS RECEIVABLE: 0.00 (-30) 0.00 (31-60) 0.00 (61-90) 0.00 (91-120) 0.00 (+)

( ) BILL COSTS AND FEES ( ) DO NOT BILL  
( ) BILL FEES ONLY ( ) CLOSE FILE  
( ) BILL COSTS ONLY ( ) FINAL BILL